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X-RAY DETERMINATION OF TEXTURE AND RESIDUAL STRESS IN LOW CONTRACTION ELECTROLYTIC CHROMIUM DEPOSITION

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13. ABSTRACT *(Maximum 200 words)*

Residual stresses are believed to be responsible for the intrinsic cracks observed in electrolytic chromium coatings. The cracks directly affect the wear and erosion behavior of the coating and substrate. Crystalline orientation significantly influences the elastic-plastic properties of materials. It also affects the method by which residual stress can be determined using x-ray diffraction. For this study, we investigated texture and residual stress analysis for two low contraction (LC) chromium coating specimens and compared the results with a high contraction (HC) chromium specimen. High-resolution pole figure analysis and x-ray diffraction were used to characterize the texture in the coatings. Randomly oriented materials allow the application of the x-ray diffraction $\sin^2\Psi$ stress measurement method. For highly textured body-centered-cubic crystals, the $\sin^2\Psi$ method failed, so a Matlab matrix inversion method was used to determine residual stress. One of the LC chromium specimens exhibited near random orientation with very weak fiber texture, and the other specimen exhibited intermediate mixed <111> and <211> fiber texture. The HC chromium specimen exhibited strong predominately <111> fiber texture. A correlation between residual stress and texture was found. The HC chromium specimen with high fiber texture showed higher surface tensile stresses, while the LC chromium specimens with more randomly oriented crystallites showed lower residual stresses.

14. SUBJECT TERMS

Chromium, Low Contraction Chromium, High Contraction Chromium, Texture, Residual Stress

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TABLE OF CONTENTS

TABLES

LIST OF ILLUSTRATIONS

INTRODUCTION

Electrolytic chromium has excellent properties, including:

- A high melting point at 1875°C, compared to substrate steel at 1538°C
- High hardness 800 to 1000 KHN_{50}, compared to steel R_C 36 to 38 (360 to 380 KHN)
- Low coefficient of friction
- Excellent adhesion to substrate steel
- An elastic modulus at 36×10^6 psi, compared to steel at 29×10^6 psi
- Inert to aggressive propellant gases

Electrolytic chromium has been the choice material to protect gun bores against high temperature wear and erosion. However, high contraction (HC) chromium coatings are known for intrinsic characteristic cracks, due to the buildup of high residual stresses causing the crystallites to coalesce. Improved low contraction (LC) chromium with fewer cracks has been under investigation (refs 1-3). Martyak and Weil (ref 4) reported an epitaxial relation between thin chromium deposits and copper and nickel substrates, and <111> fiber texture as the chromium deposit thickness increased. Our analysis showed <111> surface fiber texture in production HC chromium, but near random surface texture in LC chromium (ref 5).

Texture affects residual stress determination of materials in several ways: texture renders many reflections unavailable for stress measurement, texture causes a nonlinear relation in *d*-spacing *versus* $\sin^2\psi$, and texture gradients can make stress measurement difficult. High tensile residual stress in HC chromium was reported by Pina *et al.* and Cassagne *et al.* (refs 6,7) assuming Kroner-Eshelby and Reuss models. Janda and Stefan (ref 8) reported stress measurements of chromium deposition on a thin circular plate. We reported high tensile stresses in laboratory HC specimens, but lower tensile stresses in production HC specimens, using a matrix inversion method (ref 9). This method was based on Clemens and Bain (ref 10), and simultaneously solved for unstrained latticed parameter and residual stress in highly textured thin films. Texture was accounted for explicitly. A modified $\sin^2\psi$ technique was also used to obtain x-ray residual stresses using multiple types of radiation and multiple families of reflection (refs 6,9).

This work investigated the texture and residual stress state of two production LC chromium specimens deposited at 85°C onto the bore of a large-diameter steel cylinder: LC-A was deposited at high current density, and LC-B was deposited at half the current density used for LC-A. Crystalline structure in chromium electrodeposition was investigated using a Scintag PTS diffractometer and locally developed quantitative high-resolution pole figure software. Residual stress measurements were conducted on a TEC stress analyzer. The $\sin^2\psi$ method gave good results in near randomly oriented LC-A. In LC-B, x-ray measurements suffered from larger errors due to the presence of texture. A laboratory LC chromium specimen was also deposited onto a brass plate, and the radius-of-curvature method was used to determine residual stress.

CRYSTALLINE TEXTURE OF LOW CONTRACTION CHROMIUM

Figure 1 shows the x-ray diffraction patterns using copper K-α radiation for the two LC chromium specimens, LC-A and LC-B, compared to HC chromium and the International Center for Data Diffraction (ICDD) database for chromium. For LC-A, deposited at high bath temperature and high current density, all reflections had relative intensities near that of the random powder. Weak preferred [200] and [211] orientations were also observed. For LC-B, all reflections were observed, with strong preferred [211] and [111] orientation, and weak preferred [310] orientation. For HC chromium, a very strong preferred [111] orientation and a broadened diffraction peak were observed.

Figure 2 shows (110) pole figures and compares LC-A, LC-B, and HC from $\chi = 0$ to 80°. The figure also shows the ¾ cut-off cross sections. For LC-A, a broad and diffused pole figure was observed, disclosing near random texture. The weak ring around $40^{\circ}\chi$ was due to the very weak preferred (200) and (211) crystallites. For LC-B, two fiber texture states, <211> and <111>, were observed. The two texture states were not well resolved, showing a broadened pole figure extending to high χ-tilts. For HC chromium, sharper <111> fiber texture was observed with good in-plane azimuthal symmetry.

RESIDUAL STRESS IN LOW CONTRACTION CHROMIUM

Due to the strong texture of HC chromium, few reflections in the high 2θ range were available for stress analysis. Residual stress was solved explicitly based on the Reuss model using a single family of reflection (ref 10). This method is applicable to cubic crystals when all of the crystallites favor one particular crystallographic orientation. Assuming an elastic isotropic model, residual stress was also determined using multiple radiation and multiple reflections. A near linear *d*-spacing *versus* $\sin^2\psi$ curve was obtained, using only a few available data points (refs 6,9).

Near random texture existed in LC-A, and intermediate texture existed in LC-B. Residual stresses in the LC-A and LC-B were analyzed using the chromium (211) reflection at 153.26° 2θ using chromium radiation. These residual stresses were determined from 31 data points as shown in Figures 3 and 4. The near randomly oriented LC-A gave a good linear *d*-spacing *versus* $\sin^2\psi$ curve. The intermediately textured LC-B showed the influence of texture–both by the reduction of intensities at certain ψ-tilts, and by the nonlinearity in the $\sin^2\psi$ curve. Crystallite orientation distribution function (ODF) needs to be considered to improve residual stress determination (ref 11). Figure 5 shows that for HC chromium, the $\sin^2\psi$ method failed completely. The positive psi angles gave an erroneous compressive stress of approximately -845 Ksi, and the negative psi angles gave erroneous tensile stresses of similar magnitude. Thus, residual stress was determined using a Matlab inversion method. A full-width,half-maximum analysis was performed for the (222) diffraction peaks, resulting in HC (2.66°), LC-A (1.85°), and LC-B (1.26°).

CONCLUSIONS

- X-ray determination of texture and residual stress results are summarized in Table 1.

- The HC chromium specimen deposited at a low temperature of 55°C and low current density exhibited strong <111> fiber texture (ref 5). Higher surface tensile residual stresses were observed using a Matlab matrix inversion method.

- LC chromium specimens were deposited at a high temperature of 85°C: LC-A deposited at high current density exhibited near random orientation, with very weak <100> and <211> fiber texture; LC-B deposited at half the current density exhibited mixed <111> and <211> fiber textures and a very weak <310> fiber texture.

- Lower bi-axial tensile residual stresses were detected in the two LC specimens. Good residual stress measurements were achieved for near randomly oriented LC-A. For LC-B, x-ray residual stress measurements were low, suffering from large errors due to crystalline texture.

- A correlation was made between the degree of texture and residual stress. Highly textured HC chromium had higher tensile residual stress compared to more randomly oriented LC chromium, which had lower residual stresses. The stresses are believed to be responsible for the cracks observed, which directly affected the wear and erosion behavior of these coatings.

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Table 1. Texture and Residual Stress in HC and LC Chromium Specimens

	Immersion HC	**Flow-Through LC-A**	**Flow-Through LC-B**
Preferred Orientation	(111)	None	(211) and (111)
Texture	Strong Fiber	Weak Fiber	Weak Mixed Fiber
Method of Stress Determination	Reuss Matrix Inversion	$\sin^2\Psi$ Method	$\sin^2\Psi$ Method
Residual Stress (MPa)	~315	133 ±10	167 ±27

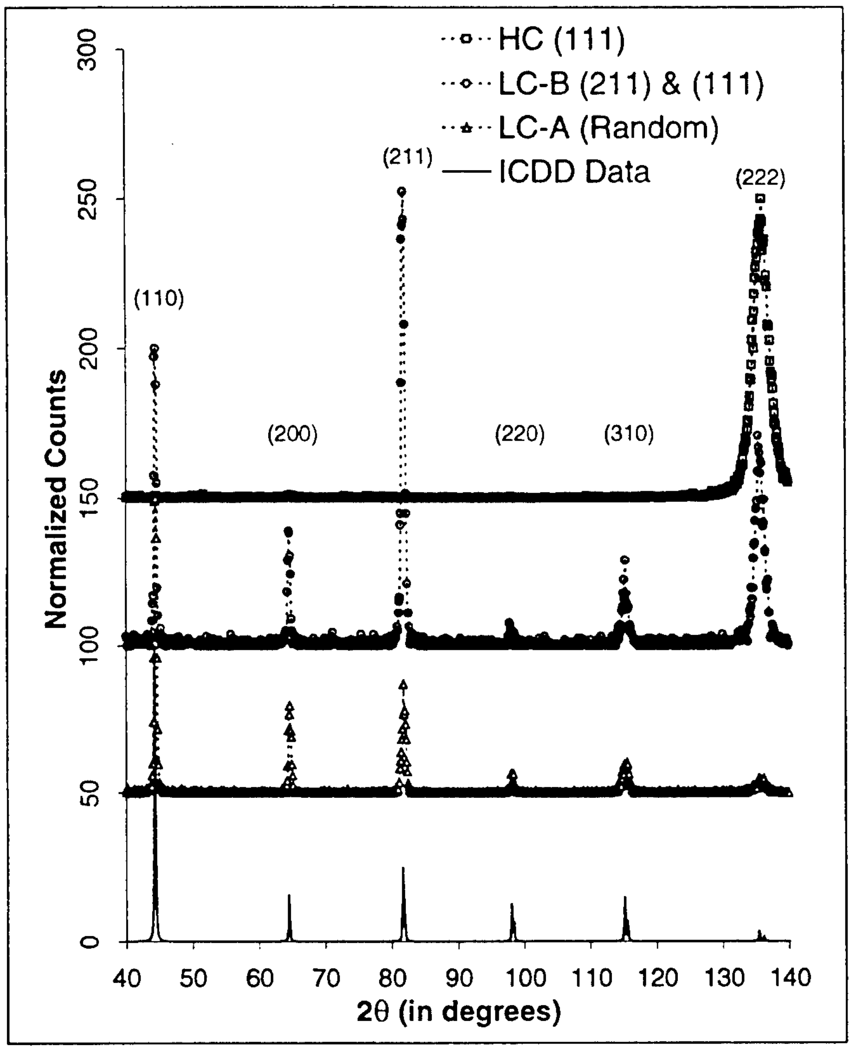


Figure 1. X-ray diffraction patterns comparing intensities of HC, LC-A, and LC-B.

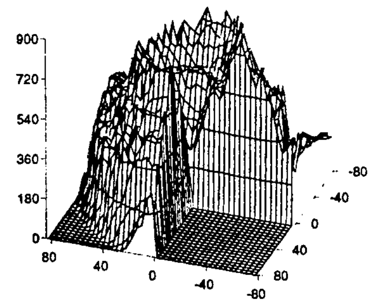
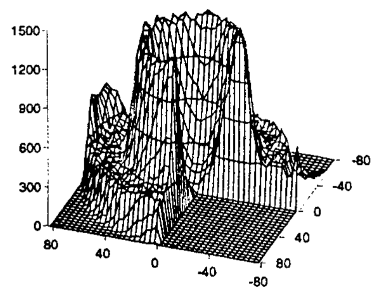
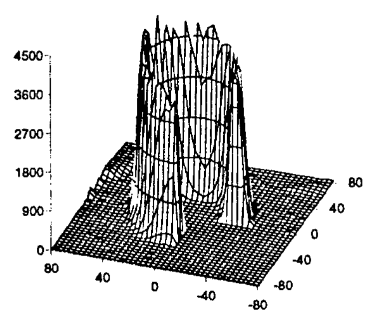
	LC-A (110) Pole Figure
Random	
	LC-B (110) Pole Figure
(110) Intercepts: [111] at angle 35.25° [211] at angles 30°, 54.75°, 73.25°	
	HC (110) Pole Figure
(110) Intercepts: [111] at angle 35.25°	

Figure 2. Chromium on steel (110) pole figures.

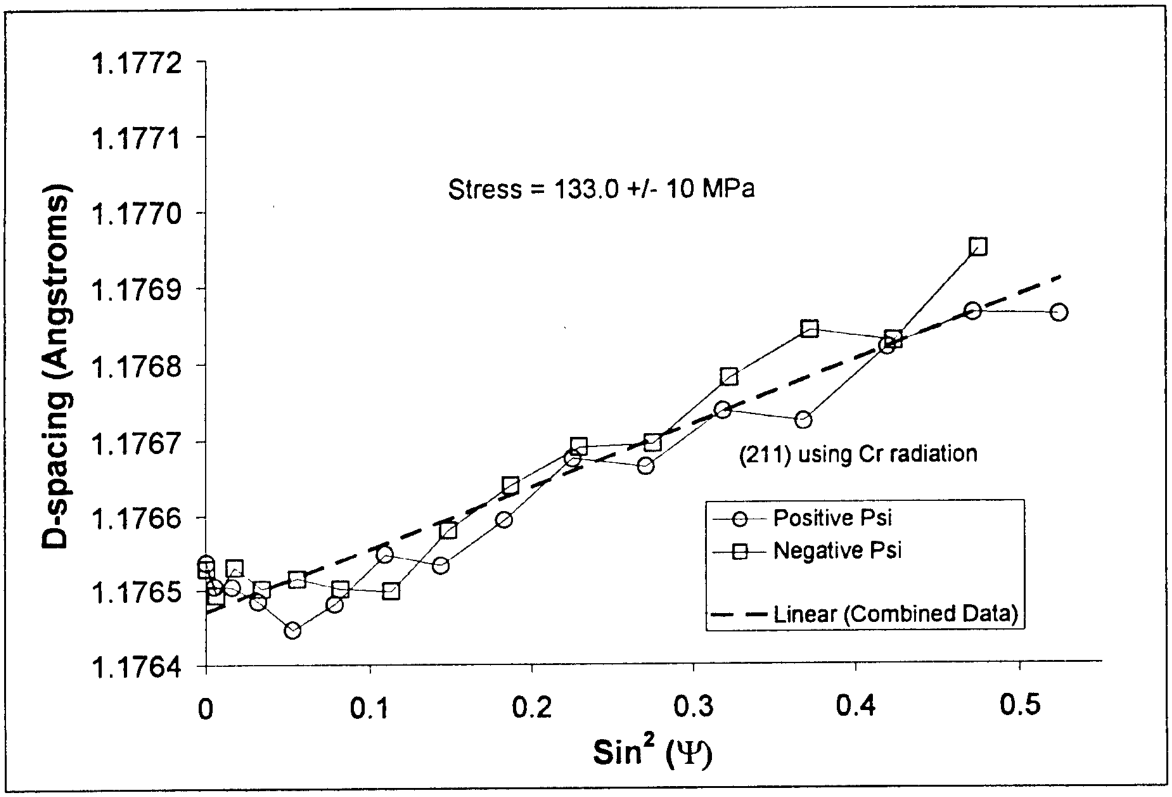


Figure 3. Residual stress in LC-A chromium coating.

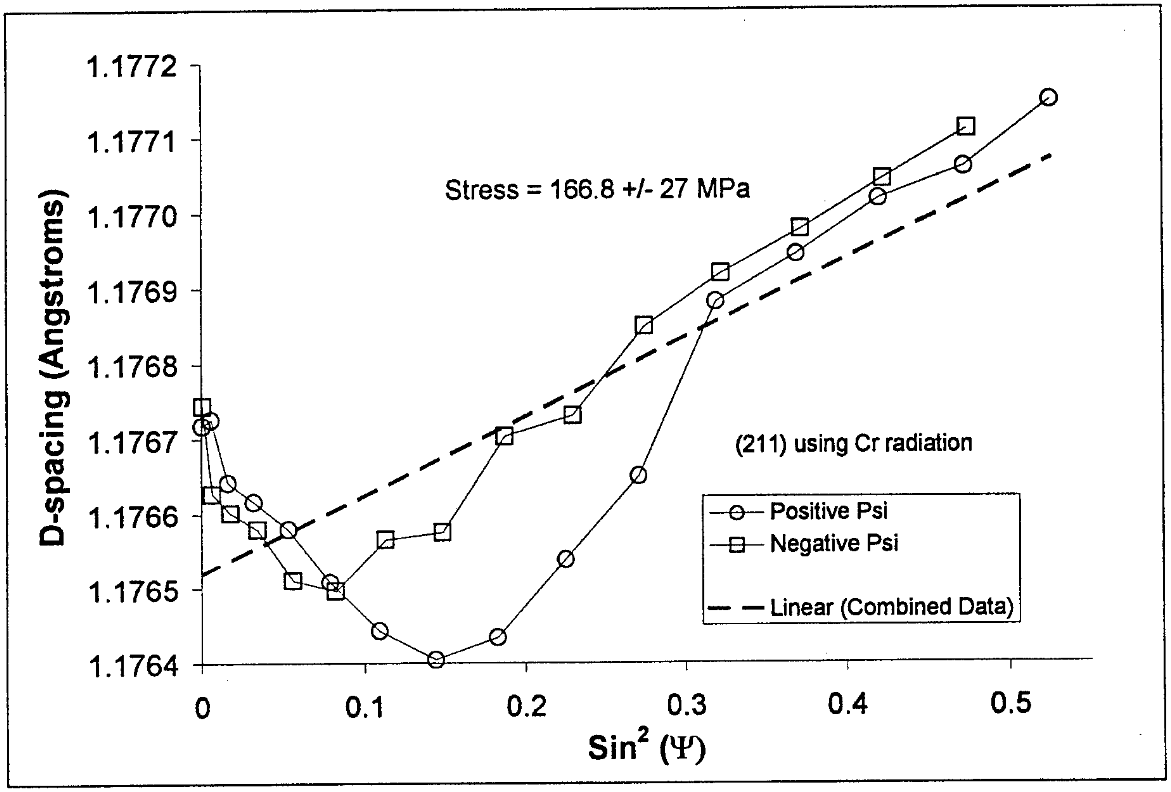


Figure 4. Residual stress in LC-B chromium coating.

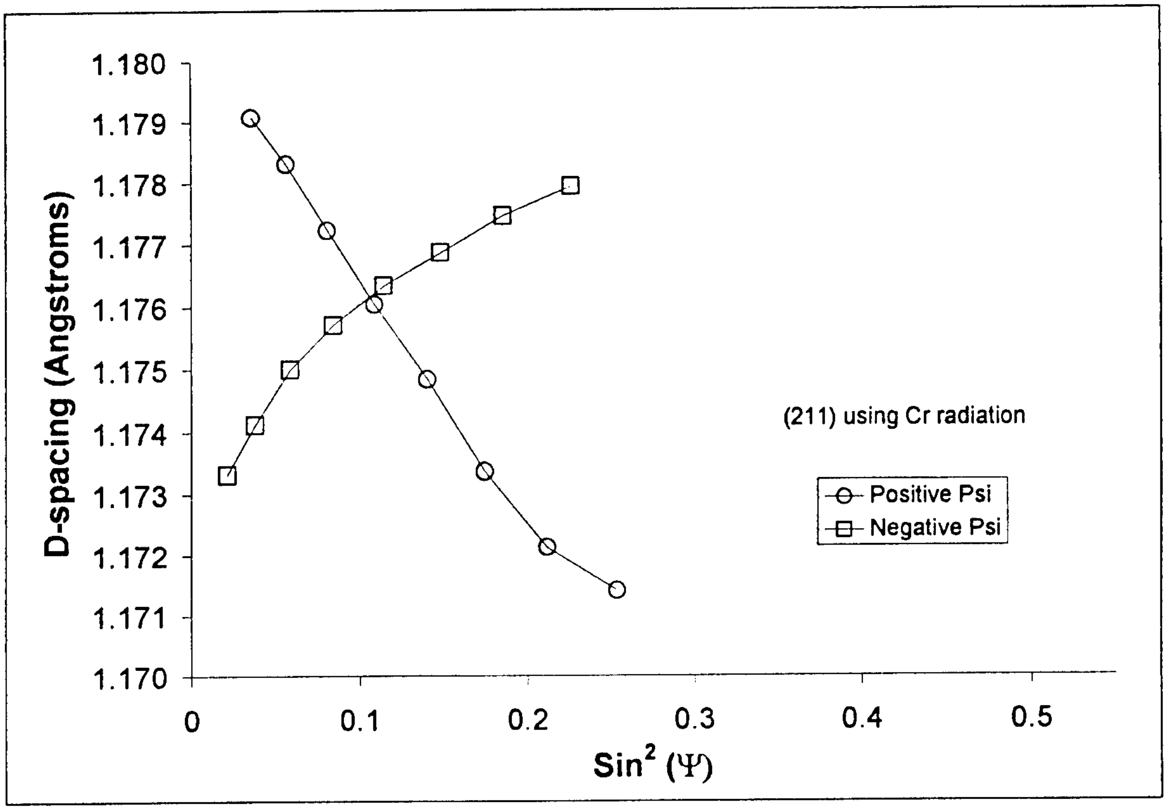


Figure 5. Residual stress in HC chromium coating.

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